(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
10 October 2002 (10.10.2002)

PCT

(10) International Publication Number
WO 02/079770 A1

(51) International Patent Classification[7]: G01N 27/02, 27/22, G01F 23/26

(21) International Application Number: PCT/NO02/00130

(22) International Filing Date: 2 April 2002 (02.04.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2001 1620 29 March 2001 (29.03.2001) NO

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(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RU, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

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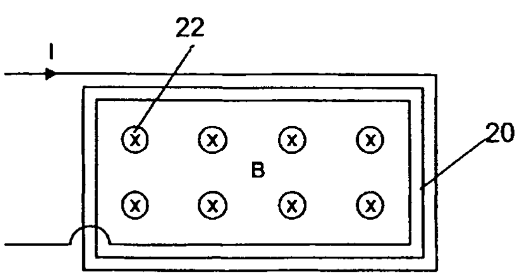
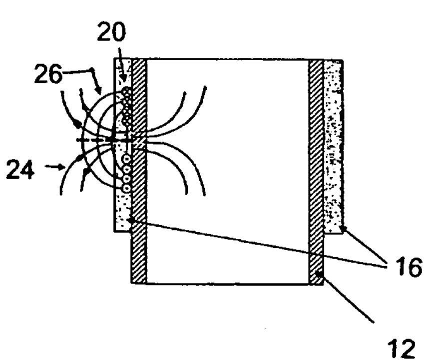
(54) Title: METHOD AND DEVICE FOR MONITORING CHEMICAL REACTIONS OR LEVELS OF A SEPARATION TANK



(57) Abstract: A method and a device for monitoring chemical conversions or reactions in fluids or particulate materials, or levels of separation tanks are disclosed. The method is characterized in measuring permittivity and/or permeability by means of an ortogonal magnetic field coil. Preferably the coil is arranged in the same plane.



WO 02/079770 A1

Published:
— *with international search report*

METHOD AND DEVICE FOR MONITORING CHEMICAL REACTIONS OR LEVELS OF A SEPARATION TANK.

The present invention relates to a method and a device for monitoring levels of separation tanks, or chemical conversions or reactions in fluids or particulate material, as defined in the preamble of the following independent claims directed to a method and a device, respectively.

The invention also relates to applications of the method and the device mentioned above.

More specific the present invention relates to the monitoring or measurements of the chemical conversions occuring when zinc oxide (ZnO) is exposed to sulfur containing substanses or substances, such as sulfur dioxide.

In several processes a catalyst bed of particulate zinc oxide ZnO is used to remove sulfide from gases.

In this context the conversion from the oxide state and into Zing-sulfur state, may be denoted as the rate of saturation of zinc oxide.

When studying a filter catalyst bed of particulate zinc oxide one considers that when the zinc oxide at the exit end of the filter has been converted into a zinc-sulphide-compound, then the catalyst filter must be replaced. In other words: the more this ZnO-conversion occurs, the closer to the filter bed exit end the interface between the ZnO level and zinc-sulphur compound level is

positioned. If the catalys bed is not replaced, then the sulfur compund wil pass throught the catalyst bed and then pollute the apmoshere.

The above explanations as to how the sulfur reacts with ZnO, should not be considered as limiting to the scope of the invention.

It is an object of the present invention to provide for a method and a device to more closely control the extent of the abovemention conversion/reaction in such catalyst beds.

Further, it is an object to provide for a new structure of a device for monitoring for example levels of contents (e.g. water/oil/gas) of separation tanks, or for monitoring the development of chemical reactions.

In other words it is a main object to more closely determine the time for replacing the ZnO catalyst bed filter material, in order to minimize the sulfur contamination of the environment.

The method of the present invention is characterized by measuring the permittivity and/or permeability by means of an ortogonal magnetic field coil (i.e. ortogonal to the dip-stick axis).

The further features appear in method claims 2-13.

The device of the present invention is characterized by the features appearing in the independent device claim 14. The preferred embodiments are disclosed in the dependent device claims 15-20.

The preferred use features are stated in claims 21 and 22.

The invention is to be explained in detail with reference to the drawing figures, in which:

Figure 1 is model of the coil structure.

Figure 2 shows a circular coil structure according to the well known structure.

Figure 3 shows a front view of an ortogonal magnetic field coil according to the invention.

Figure 4 shows a cross section of the ortogonal magnetic field coil according to the invention, and installed on a test dip-pipe.

Figure 5 shows a cross section of a vertical coil installed on a dip-pipe.

Figure 6 shows a principle of a level point detector coil compared to a reference coil.

There are many principles for determining levels of liquids or solids in tanks and can be based on capacitance, microwave, nuclear radiation etc.

High frequency magnetic coils developed to measure the water, oil and gas interfaces in gravitation separators are previously known.

n a straight conductor a magnetic field is generated, due to the alternating current flow through the conductor. The magnetic field is 90° to the direction of the current and thus the magnetic field is circular around the conductor. If the conductor is wound to a coil the magnetic field is strongest in the centre of the coil (see figure 2).

The high frequency magnetic coil is sensitive to dielectric, inductive and conductive properties of surrounding materials. This make this principle well suited for many applications, where materials have different conductivity, permeability and/or permittivity. One example of applications is water-oil-gas interface determination in separators. By using high frequencies, higher spacial resolution is obtained due to the low number of windings needed.

In general, the model of a coil is an inductor L and a capacitance C in parallel, as shown in the enclosed Figure 1. The contributions of the overall coil capacitance include capacitances between the windings, capacitance between the coil and the surrounding material, and stray capacitances in cables etc.

When the high frequency magnetic coil is dipped into a material, for example a fluid, its electrical properties will change. If the material is electrical conductive the

inductance of the coil will decrease due to the eddy current induced in the fluid. And if the permittivity is changed, the coil capacitance is changed. One or more coils can be installed on a non-conducting dip-pipe, depending on the application. For a profiler with a certain resolution, closed stacked coils are installed.

The capacitances between the windings of the coil are fixed and related to the coil diameter, the wire diameter and the number of windings of the coil. Stray capacitances are minimized by installing the electronics closed to the coil, i.e. on the inside of the dip-pipe. The capacitance between the coil and the surrounding material depends on the electrical permittivity of the fluid. This means the coil is sensitive for permittivity as well as permeability properties of the surrounding material.

The coil is connected to a LC-oscillator, where the resonance frequency of the coil is:

$$\omega = \frac{1}{\sqrt{LC_L}} \qquad (1)$$

where L and C_L are the inductance and equivalent parallel capacitance of the coil, respectively.

Different surrounding materials cause different resonance frequencies. Thus, interface levels can be predicted. The equivalent coil capacitance will depend on the electrical permittivity of the surrounding material and therefore influence on the resonance frequency.

In order to measure the different levels of for example water, oil and gas in a separator tank, a dip-pipe is submerged in the tank so that the distribution of the different phases covers the dip-pipe. A number of coils are mounted on the dip-pipe, at mutual intervals. The distance between the coils is selected according to the adequate accuracy of the measurements. The sensitivity of the measurements is increased by using high frequency magnetic fields in the range of 5-25 MHz (mega hertz). It is therefore not possible to use iron cores, as the frequency

of the magnetic field consequently is limited to about 2 MHz. The high frequency also restricts the number of windings of the coil, and the distance between the coil and the electronics. In order to use such high frequencies, the electronics must be arranged inside the dip-pipe, just behind the coil, so that the distance between the coil and electronics is at minimum.

Thus the measuring area is outside the dip-pipe. Previously the coils are wounded around the dip-pipe, but this gives the strongest magnetic field in the centre of the dip-pipe, and the weakest in the measuring area, outside the dip-pipe, as illustrated on figure 2. This kind of coil is denoted as a circular coil. In general a coil consists of inductance and capacitance in parallel as shown in figure 1. When circular coils are mounted on a dip-pipe without the coils being protected against mechanical wear and tear, the circular coils are sensitive to changes in the permittivity (capacitance) of the surrounding medium. The high stray capacity between the the windings and the medium is the reason for this sensitivity. An electric field is directed from the coil windings and into the medium. In a separator containing water, oil and gas, all three components may be indentified, and also the position of the level between the non-conducting oil and gas phases.

When a coil 10 is installed at the circumference of a dip-pipe 12, as shown in Figure 2, the magnetic field 14 is directed vertically. The magnetic field will be weakest on the outside of the dip-pipe, where the material of interest is. That means the changes due to permeability and conductivity of the surroundings have a minor effect on the detector. Further, due to necessary mechanical protection of the coil, the influence of changes in the permittivity of the surrounding material is reduced.

Thus, this circular coil is not well suited in the level prolifer.

To increase the sensitivity to changes due to permeability, conductivity and permittivity of surrounding materials by using coils with insulation, the coil is

structured so that the magnetic field is directed ortogonally to the dip-stick axis and into the surrounding material. The resonance frequency of this ortogonally field coil is also derived from Eq 1.

In Figure 3, the front view of the coil 20 is shown, and the magnetic field 22 is directed into the paper plane. The coil conductors are wound o form a coil in the same plane (flat or even). As shown in the figure, the coil 20 is wound in square shaped loopes in the same plane. Other configurations in the same plane are also feasible. For example the plane may be curved for the coil assembly to cover the outer surface of a circular dip-pipe. The shown coil configuration 20 gives a high magnetic field 24 directed into the material to be measured. Further, according to the Maxwell equations, the electrical field 26 is always perpendicular to the magnetic field lines 24. This means there are capacitances between the coils, following the electrical field lines, as shown in Figure 4. Due to the extension of the magnetic field 24 into the material, the penetration of the electrical field 26 is considerable. Therefore, this coil 20 is sensitive for changes in dielectric and conductive properties in materials even if the coil 20 is insulated outside by means of an insulation layer 16. Optionally the coil with its plane is bent into an arc form, in order to fit the outside of a circular hollow pipe. Then the coil is embedded in a matrix of a plastic of resin 16.

A problem by using conventional capacitive sensors is its high sensitivity to deposits on the electrodes, thus the changes in sensitivity will occur. Due to the deep penetrating electrical field, this sensor offers a low sensitivity to deposits, like scaling, waxing etc.

Conventional capacitive sensors used in the oil-industry are not suitable, since it will not function in water-continuous mixtures. The high frequency magnetic, however, will work in both oil-continuous and water-continuous mixtures.

With reference to figure 4, the coil is embedded in an insulation layer material 16. The insulation material may comprice a plastic or resin material, said materials being known in the art. The thickness of the insulation layer is preferably in the range of 2-10 mm (millimeters), and preferably in the range of 3-6 mm. The device according to the invention has been tested sucessfully with protection layer thicknesses of 3 and 6 mm, respectively.

The dip-pipe according to the invention may consist of an extended hollow rod where a plurality of individual coils (as shown on figures 3 and 4) are mounted (as closed stacked coils) to the outer surface of the rod as according to the invention, and thereafter covered by the protective insulation layer. Each coil unit is connected to the electronics which is positioned inside the dip-pipe/rod. The result of the measurements from each coil assembly is one at a time extracted and handled by the electronics.

In an industrial environment, the coils must be protected mechanically with an insulation material, for the magnetic field to penetrate into the surrounding medium. However, with this insulation material, the circular coils loose sensitivity to changes of permittivity, and are almost useless for measurements of oil and gas or Zno/ZnS-pellets which constitutes a part of the present invention. But the coils are still suitable for measurements based on inductive changes, such as between oil and water.

If the coil is turned 90° in relation to the dip-pipe, as according to the present invention, the magnetic field (at 22) in the centre of the coil will be directed into the medium, instead of along the longitudinal direction of the dip-pipe. Now, the strongest magnetic field of the coil is directed straight into the medium. As the current is constant, the density of the magnetic field lines remains unchanged. If the coil is narrowed or squeezed so that the height is smaller than the width/length of the coil, as illustrated on figure 3, the density of the magnetic field lines of the passage between the upper and lower portion of the coil 20, increases. Figure 4 shows a cross section of

the coil 20. The increased density of field lines means that the magnetic field lines 24 go farther from the coil 20 itself, and deeper into the surrounding medium. As the magnetic field 24 goes deeper into the medium, also the electrical field 26, which is directed perpendicular to the magnetic field lines 24, penetrates deeper into the medium.

The electrical field lines 26 are directed between two conducting materials. This means that the longer the electrical field penetrates the medium, the larger is the sensitivity of the coil to changes of permittivity. This means that the coil can be insulated using a mechanical protection, at the same time maintaining the sensitivity to changes of permittivity. Figure 4 shows the electrical field lines 26 penetrating the medium even though the coil 20 includes a protection layer 16. In the experiments being conducted, protection layers with a thickness of 3 mm (millimeters) and 6 mm, have been used.

When utilizing the method and the coil device according to the invention, high frequencies are used. In this regard, high frequency means frequencies in the range of 2-30 Mhz, and preferably in the range of 5-25 Mhz. As will appear from the following Examples, resonance frequncies in the range of 10-11, are used.

<u>Use of invention in relation to measurements of catalyst beds of ZnO. Rate of saturation, i.e conversion of ZnO to ZnS</u>.

The permittivities of pure ZnO (pellets) and ZnS their pure state, are ca 2 and ca 8, respectively. The pellets are mainly spherical, thus the measured permittivity will be an average permittivity of air and pellets. Said resulting permittivities are 3.1 and 4.3 respectively. The coil according to the invention, can be used to determine when the surrounding material turns from the ZnO to the zinc-sulphide compound state, i.e. one may establish as to when the pellets are saturated or consumed, for example at the catalyst bed exit end, and the pellets of the catalyst bed must be replaced.

Measurements have been done to determine said average permittivities of 3,1 for Zno and 4,3 for ZnS, respectively. The difference can result in a maximum frequency shift of about 10%. Due to coil insulation, the capacitance of the input stage of the electronics etc, the frequency shift will be considerably reduced. Measurements show that this frequency shift can be more than about 2,5.

It is assumed that in operation, an interface level between ZnO and ZnS is moving upwards as the reaction between ZnO and the sulfur gas is proceeding. By using a series of coils according to the invention, it is possible to establish the position of said interface level.

Thus the present method may be used to control chemical changes of any substances between two or more chemical states. In other words the present method and device may be used in relation to any materials changing permittivity and/or conductivity when reacting.

With reference to figures 3 to 6, ortogonal magnetic field coils installed on a dip-pipe, were tested. The advantage of these coils is that the magnetic field is directed ortogonally of the dip pipe out into the surrounding media. Thus a stronger magnetic field is achieved in the surrounding media compared to that of circular coils. In circular coils (as shown in figure 2) the magnetic field is strongest in the middle of the dip pipe. In addition to the number of turns, the length of the coil, i.e. the angle α (see figure 5) influences the sensitivity of the coil as shown i table 1.

Used in catalyst beds, there will be no need for waterproof insulation, and experiments including thinner types of insulation, were done. Results from these initial measurements are presented in table 1. As shown the frequency shift is relative low, 2,3%, related to the oscillation frequencies. Therefore, when one coil is used as a level gauge 40 (see figure 6), a reference coil 50 located in ZnO can be used to give more accurate measurements. The level gauge detector coil 40 is matched

with the reference coil when the catalyst bed is refilled with ZnO, as shown on figure 6.

By multiplying the two sinusoidal signals from the oscillators 42,52, the difference between the frequencies can be obtained by filtering the output signal. When this output is low (zero) and constant, the materials surrounding the coils, are equal. When the output signal starts to increase, the surrounding material is changing, and with a constant high output signal, the surrounding materials are different. Thus with the level gauge coil and a reference coil, a monitoring system is achieved, in order to give more information of the time to replace the pellets.

Table 1. Measurements of resonance frequency for ortogonal field coils in pellets.

	Resonance frequency, air [MHz]	Resonance frequency, ZnO [MHz]	Resonance frequency, ZnS [MHz]
without insulation a = 270°	11,182	10,740	10,571
0,5 mm insulation a = 270°	10,200	10,045	9,813

In these initial measurements presented here, the highest sensitivity between measurements of ZnO and ZnS (saturate ZnO) is found to be about 232 KHz. A principle set up of a catalyst bed monitoring system is shown in figur 6.

PATENT CLAIMS

1. Method for monitoring chemical conversions or reactions in fluids or particulate materials, characterized in measuring permittivity and/or permeability by means of an ortogonal magnetic field coil.

2. Method according to claim 1, characterized in using an ortogonal magnetic field coil wherein the number of turns are arranged in the same plane, e.g. the coil is arranged on a plate.

3. Method according to claims 1-2, characterized in using a coil assembly forming an arc form.

4. Method according to any of claims 1-3, characterized in using a coil arranged in an arc form extending to cover a sector of a circle.

5. Method according to any of claims 1-3, characterized in monitoring chemical conversions or reactions in fluids or particulate materials, in particular materials hich are changing permittivity and/or conductivity due to chemical reactions.

6. Method according to any of claims 1-3, characterized in monitoring chemical sulfur saturation of ZnO in a catalyst bed.

7. Method according to any of claims 1-3, characterized in monitoring the interface between pure ZnO level and SO_2 saturated ZnO level in a catalyst filter bed.

8. Method according to claim 7, characterized in using a coil as a level gauge (figure 6), and a reference coil located in ZnO is used to estimate the time for refilling the catalyst bed with ZnO.

9. Method according to claim 7, characterized in that two sinusoidal signals from the oscillators are multiplied, the difference between the frequencies being obtained by filtering the output signal so that:

when the output is low (zero) and constant, the materials surrounding the coils are equal, and when the output signal starts to increase, the surrounding material is changing, and with a constant high output signal, the surrounding materials are different.

10. Method according to any of the preceding claims, characterized in using a measuring device having a plurality of individual coils which are mounted to the outside of an extended dip-stick.

11. Method according to any of the preceding claims, characterized in that the coils are embedded in an insulating protective layer, preferably having a thickness in the range of 2-10 mm (millimeters), and preferably in the range of 3-6 mm.

12. Method according to any of the preceding claims, characterized in that insulating protective layer comprises a plastic or resin material.

13. Method according to any of the preceding claims, characterized in using high frequencies in the range of 2-30 MHz, and preferably in the range of 5-25 Mhz, and most preferably in the range of 10-11 MHz.

14. Device for monitoring chemical conversions or reactions in fluids or particulate material, characterized by an ortogonal magnetic field coil wherein the number of turns are arranged in a same plane.

15. Device according to claims 10, characterized in using a coil arranged in an arc form.

16. Device according to claims 9-10, characterized in using a coil arranged in an arc form extending to cover a sector of a circle.

17. Device according to any of the preceding claims 14-16, characterized in the measuring device having a plurality of individual coils which are mounted to the outside of an extended dip-stick.

18. Device according to any of the preceding claims 14-17, characterized in that the coils are embedded in an insulating protective layer, preferably having a thickness in the range of 2-10 mm (millimeters), and preferably in the range of 3-6 mm.

19. Device according to any of the preceding claims 14-18, characterized in that the insulating protective layer comprises a plastic or resin material.

20. Device according to any of the preceding claims, characterized in being arranged to use high frequencies in the range of 2-30 MHz, and preferably in the range of 5-25 MHz, and most preferably in the range of 10-11 MHz.

21. Use of the method and device according to any of the preceding claims, for monitoring the levels of contents (e.g. water/oil/gas) of separation tanks, or for monitoring the development of chemical reactions.

22. Use according to claim 21, for monitoring or measurements of the chemical conversions occuring when zinc oxide (ZnO) is exposed to sulfur containing substanses or substances, such as sulfur dioxide.

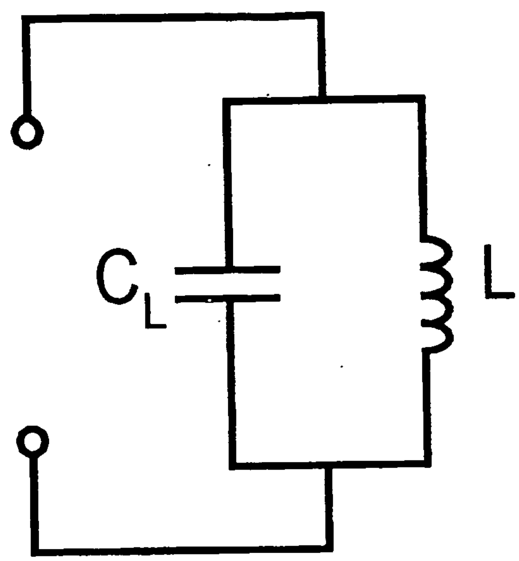


FIG 1

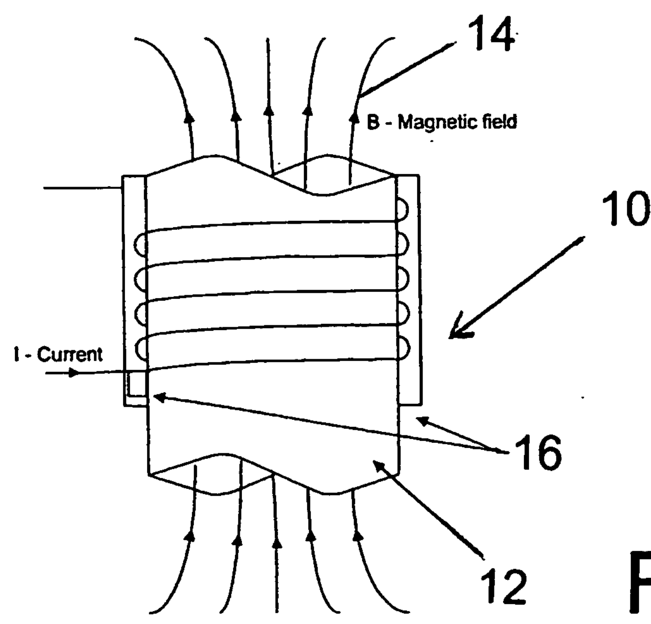


FIG 2

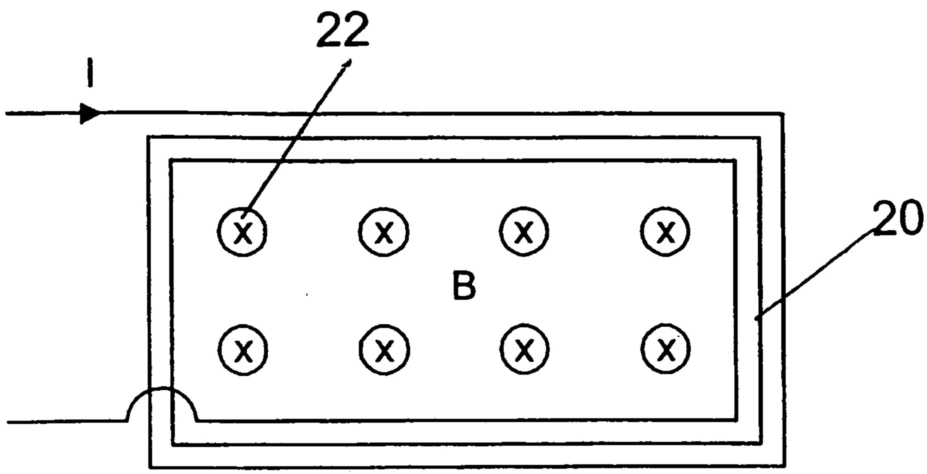


FIG 3

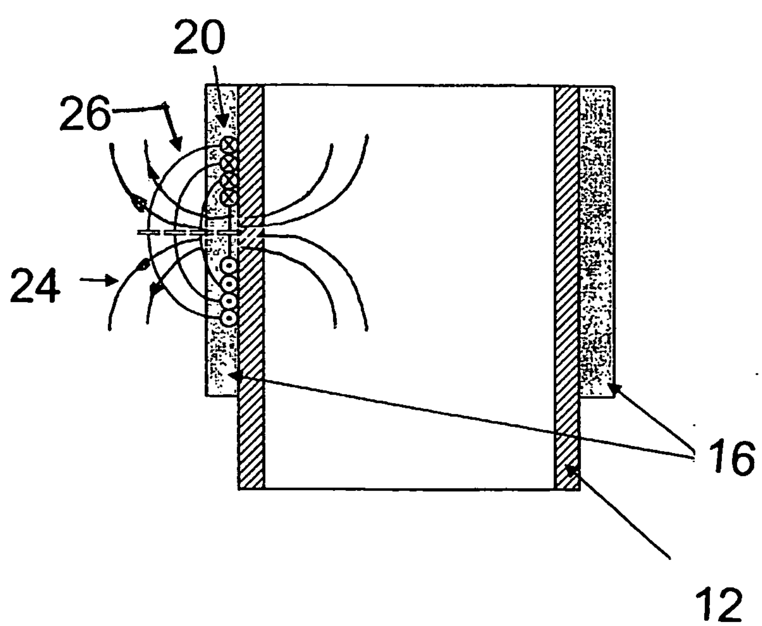


FIG 4

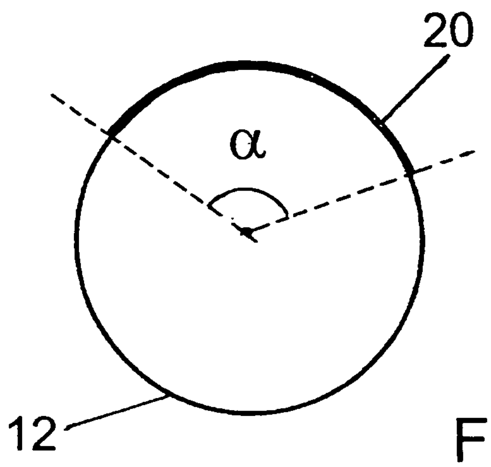


FIG 5

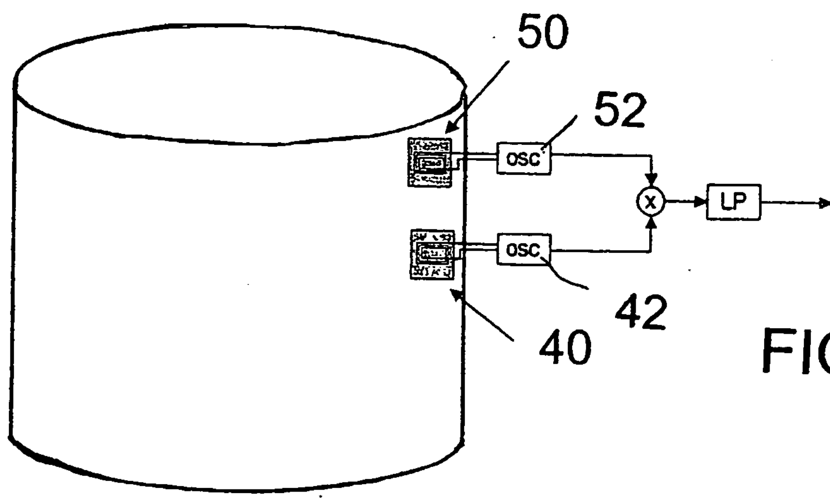


FIG 6

INTERNATIONAL SEARCH REPORT

International application No.
PCT/NO 02/00130

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: G01N 27/02, G01N 27/22, G01F 23/26
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: G01N, G01F, B22D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-INTERNAL, WPI DATA, PAJ, INSPEC

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	US 4688580 A (KO ET AL), 25 August 1987 (25.08.87), column 4, line 9 - line 14, figure 4 --	1-22
Y	US 4138888 A (LINDER), 13 February 1979 (13.02.79), column 5, line 20 - line 65; column 8, line 15 - line 48; column 9, line 15 - line 25, figures 1,2,6,8 --	1-22

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
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Date of the actual completion of the international search	Date of mailing of the international search report
2 July 2002	08-07-2002
Name and mailing address of the ISA Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Gordana Ninkovic/SN Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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INTERNATIONAL SEARCH REPORT

Information on patent family members

10/06/02

International application No.
PCT/NO 02/00130

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				IN	171236	A	22/08/92
				IT	1222337	B	05/09/90
				IT	8783479	D	00/00/00
				US	4893507	A	16/01/90
DE	3423868	A1	02/01/86	NONE			